

The Measurement of the Properties of Materials

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Invited by G. W. CHANTRY

Invited Paper

This review covers approximately 15 years of development in the techniques used to measure dielectric properties of materials over the frequency range 1 MHz to 1500 GHz. An introductory section summarizes the broad development trends and is followed by short sections which deal with developments at a more detailed level. The approaches described include time- and frequency-domain methods; reflection, transmission, and resonant methods; guided free-space methods; discrete-frequency and broad-band methods; especially Fourier Transform Spectroscopy. Measurements on magnetic materials are also briefly discussed.

INTRODUCTION

Most of the measurements made on materials at radio frequencies and above are of their dielectric properties, magnetic measurements being rather rare. This review will therefore be concerned mostly with dielectrics, but magnetics will be briefly mentioned at the end. At radio frequencies one can actually determine, via phase-shift and attenuation measurements, the two components, permittivity and loss ϵ'' , of the complex relative permittivity $\hat{\epsilon} = \epsilon' + i\epsilon''$ where i is the square root of -1 , whereas at optical frequencies one measures instead the two components, refractive index n and absorption coefficient α of the complex refractive index $\hat{n} = n + i\alpha/4\pi\nu$ where ν , the wavenumber, is given by the frequency ν divided by the speed of light in vacuum c . The two practices are however quite equivalent and indeed even in the general case where the medium may also be magnetic and conducting one can always relate the various quantities by the use of the

Maxwell relation

$$\hat{n} = c[\mu_0\hat{\mu}(\epsilon_0\hat{\epsilon} + i\hat{\sigma}/2\pi\nu)]^{1/2} \quad (1)$$

where μ_0 and ϵ_0 are, respectively, the permeability and permittivity of free space, $\hat{\mu}$ is the complex relative permeability, and $\hat{\sigma}$ is the complex conductivity. However, for the case of nonmagnetic materials without free charges (1) reduces to the simple relation

$$\hat{n}^2 = \hat{\epsilon} \quad (2)$$

since $c = (\mu_0\epsilon_0)^{-1/2}$.

Dielectric measurements may be made for purely scientific purposes, e.g., in connection with the investigation of relaxation phenomena [1], but they are nowadays of increasing importance in telecommunication and in the design and specification of circuit components and quasi-optical elements. The latter two categories would include insulators, supports, beam dividers, lenses, resonators, substrates, windows, dielectric waveguides, radomes, radiation-absorbing materials (RAM), etc. For all these applications one needs high precision in the measurement. In some applications, such as the construction of microwave ovens, diathermy applicators, and industrial dielectric heating applicators, the dielectric loss is a critical parameter. The magnetic quantities are of crucial importance in the design of isolators, circulators, and filters. At the lower end of the frequency range one has both single-frequency and broad-band methods available and the same is true of the upper end now that CW laser sources have been developed to complement the established technique [2]–[6] of dispersive Fourier transform spectrometry (DFTS). In the middle range, that is, the millimeter-wave band, most of the measurements have been done solely by DFTS. Significant contributions have come in recent years from the groups at NPL, Teddington, U.K. [5]–[8] and the Magnet Laboratory at MIT, Cambridge, MA, USA [9]–[11]. The difficulties of making measurements on a wide range of materials over a wide

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frequency (and sometimes temperature) range have led to the development in tandem, not only of direct methods, as described so far, but also of indirect methods. In the latter other quantities, such as periodicity of a channeled spectrum, variation of transmission with angle of incidence, reflectivity, etc., are measured and these are used then to give the underlying optical parameters. It goes without saying that the direct methods give the better accuracy, but for some engineering applications and for the more difficult scientific cases, e.g., nearly opaque specimens, the indirect methods can nevertheless give results of acceptable accuracy.

At microwave frequencies and below, the direct single-frequency methods, usually involving a cavity, open resonator, or transmission line to enhance sensitivity, have had to contend in recent years with the more convenient, quicker broad-band methods, either swept-frequency instruments or else the Time-Domain Spectrometer (TDS) (see Section II-B). During the 1970s, the application of DFTS, TDS, and of other broad-band techniques was pushed toward the mid-gigahertz frequencies. Thus TDS can now operate over 5 decades with an upper limit in the 20-GHz region, while DFTS techniques are used down to 60 GHz [9]. Continuous frequency coverage is also necessary for technical reasons. For instance, it used to be argued that dielectric behavior below 100 GHz was dominated by slowly varying relaxations which allow many materials to be adequately characterized at a few frequencies at octave intervals, but this argument does not necessarily apply to lossy, composite, biological, or magnetic materials, all of which are assuming greater importance in microwave engineering.

In spite of the development of broad-band methods, it is certainly not the case that discrete frequency and narrow-band methods have had their day. Development of such techniques, in fact, continues unabated. There are a number of reasons for this. Many applications require a degree of sensitivity which has not yet been achieved by the broad-band approaches. Measurement of low dielectric loss, for instance, is still dominated by resonant (i.e., multipass) techniques (Sections II-E and II-F). In addition, many engineering applications remain relatively narrow-band, and discrete frequency measurements are, of course, acceptable for these.

Computer control of metrological apparatus is now the norm and dielectric measurements offer no exception, but the computer is playing another role in the measurement of materials: that of allowing absolute measurements of material properties to be carried out in entirely new measurement geometries. It is now possible, for instance, to compute electromagnetic fields in heterogeneous geometries (i.e., those with two or more dielectric species present) and to use the numerical analyses directly in the metrological process, possibly in real time. Examples of this approach are the measurement of dielectric properties using open-ended coaxial lines (Section II-D) or dielectric resonators (Section II-E). The use of such computer-centered techniques is now one of the most fruitful factors in generating entirely new approaches to materials metrology.

Earlier general accounts of materials metrology which are still very much to be recommended include Von Hippel [12], [13], Bussey [14] and Fellers [15] and the *Proceedings of a Tutorial Conference on High Frequency Dielectric Mea-*

surements [16]. More recent surveys of the field include those of Lynch [17], Cook and Jones [18], Kaatz and Giese [19], Birch and Clarke [8], and Alder *et al.* [20]. Developments in DFTS are to be found in Birch and Parker [3] and Afsar [6]. The series of books *Infrared and Millimeter Waves* [21] contains many articles on materials measurement at frequencies well into the microwave range. Biological dielectric measurements which are now assuming such importance because of safety considerations are described in books by Pethig [22] and Grant *et al.* [23]. The latter also discusses a number of measurement techniques at a more general level. Details of modern applications of dielectric and other materials in microwave engineering are to be found in Laverghetta [24].

Although there is now complete overlap and coverage of the RF to infrared band it is still convenient to divide the description of the experimental methods into "microwave" methods, based on microwave type of hardware, and "optical" methods, almost, but not quite, exclusively based on free-space interferometers. The next two sections cover these in turn.

II. RF AND MICROWAVE MEASUREMENTS

A. Two-Terminal Measurements

In the lower megahertz region the classical approach to the measurement of dielectric materials treats the sample as a lossy capacitance to be measured in a bridge circuit or else resonated with an inductor. Above 1 MHz the effectiveness of screening electrodes (used in three-terminal measurements at lower frequencies) diminishes rapidly with frequency. As a result, the established two-terminal unscreened approach over 1–100 MHz, namely, the resonance substitution method originally described by Hartshorn and Ward [25], has always delivered poor uncertainties (0.5 percent or generally worse) for permittivity. However, the method has remained in favor because of its ability to measure very low dielectric losses (loss angles of 10 μ rad or less) in solids by means of accurate *Q*-factor determinations. The technique has thus been the subject of intense investigation by a number of workers as a sensitive means of measuring low-loss polymers [26]–[31].

Mathematical corrections are usually used to overcome the effects of fringing fields in two-terminal cells. Similar problems occur in the UHF analog of the two-terminal cell, the TEM re-entrant cavity. Such cavities have a long history but a number of approaches have recently been made towards a more accurate computation of the resonator fields [32], [33]. Kaczkowski and Milewski [32] estimated uncertainties of the order ± 1 percent or less for permittivity and $\pm (5 \text{ percent} + 5 \times 10^{-5})$ for loss for a wide range of materials using their computational technique.

For lossy materials, nonresonant techniques employing RF bridges are more appropriate. Two-terminal cells are used up to 100 MHz [34]. Lynch [35] has described the use of an unbalanced bridge for the measurement of dielectric loss to 100 MHz. An ingenious variable temperature bridge has been described by Pratt and Smith [36]. It effectively uses two separate (but mutually phase-locked) RF sources to balance the bridge and operates from 10^{-1} Hz to 10 MHz and from -200°C to 200°C .

B. Time-Domain Methods

In the late 1960s the combined development of fast sampling oscilloscopes together with tunnel-diode step generators having rise times as fast as 35 ps allowed traveling-wave time-domain spectrometry to be used widely for the first time [37], [38]. By 1973, when van Germert wrote a comprehensive review of time-domain materials measurement, many of the basic features had become established [39], [40]. Microwave TDS systems have since, in the main, been built around proprietary sampling oscilloscopes and step generators with purpose-built coaxial sample cells. For the study of liquids up to 10 GHz, the adoption of nondispersive (i.e., coaxial) measurement cells presents no major problem, but for solid materials machining requirements are rather more of a nuisance. Although the bulk of the literature has therefore been concerned with liquid dielectrics, techniques specific to solids have also been developed. Numerous sample geometries have been used for this work, notably those in Fig. 1(a)–(d), as discussed in recent surveys by Grant *et al.* [23] and Yu *et al.* [41].

TDS is now commonly used to 10 GHz (or above, with a sensitivity which reduces rapidly above that frequency) but the past decade has seen a number of theoretical and practical refinements to existing techniques. Fellner-Feldegg's "thin-sample" technique has been developed and improved by Springett and Bose [42], [43] and van Germert [42], [43]. For less lossy materials, "multiple reflection" TDS [with thicker samples] has been developed [44]–[47]. A simpler analysis can be achieved with geometries which view samples as being either infinitely thick (see, for instance, Bucci *et al.* [48]) or else as a lumped admittance component (Rzepecka and Stuchly, [49]). Transmission methods (as opposed to TDR) are also used [40], [50]. Many of these papers present critical comparisons of the different

methods, as does a more recent paper by Gabriel *et al.* [51]. In the area of TDS theory, Cole [52], [53] has developed the theoretical basis for viewing dielectric behavior as a purely time-domain phenomenon, rather than a Fourier transform of a frequency response (see also Chahine and Bose [54]).

Papers on instrumental refinements have been concerned with the problems of time referencing (i.e., of synchronization of the sampler and of drift and phase jitter) [55], [56] and, most noticeably in recent years, with techniques of automation [57]–[60]. Developments in automation should have a significant influence upon the continuing popularity of time-domain techniques. Of specific interest is the time-domain synthesis technique [61] which is being facilitated by modern automatic network analyzers.

Below 1 MHz, excellent performance has been obtained from purpose-built automatic "step response" systems in which the samples are measured in capacitance cells. The response is compared with that of a balancing air capacitor of equal capacitance. Hyde [62] described two such systems covering overlapping frequency bands between 10^{-4} and 10^6 Hz. They employed voltage steps of up to 90 V to increase the resolution. Mopsik [63] has more recently improved upon this technique with a system operating to 10^4 Hz, utilizing CMOS switching to achieve a resolution in loss-tangent of $10 \mu\text{rad}$ —this is much better than is associated with TDS techniques at higher frequencies.

C. Frequency-Domain Transmission Line Techniques

Time-domain methods at microwave frequencies offer broad bandwidth at the cost of resolution. For the most accurate measurements of permittivity and loss one still turns to the frequency domain (FD), particularly for low-loss measurement. Thus Lynch and Ayers [64] demonstrated that

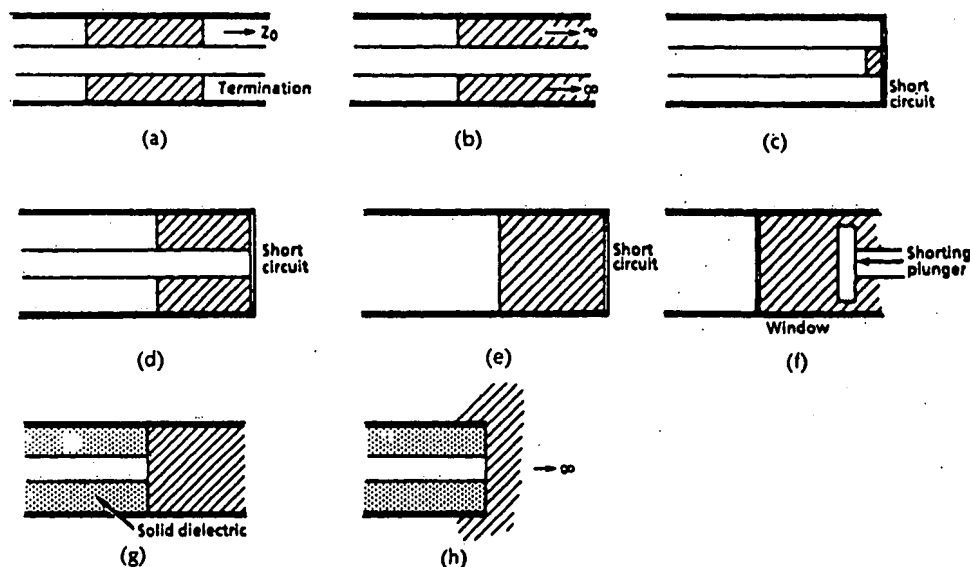


Fig. 1. A selection of sample configurations used in both time-domain and frequency-domain transmission line measurements. In each case the material under test (liquid or solid) is shown shaded. (a) Sample in matched line, for low-loss materials multiple reflections must be taken into account. (b) "Infinite sample" method for lossy dielectrics, only one reflection is taken into account. (c) Lumped admittance termination (suitable for solid samples). (d) and (e) Roberts, von Hippel geometries for dielectrics in coaxial and waveguide transmission line. (f) A variable thickness method for liquids in waveguide, (g) Discontinuous inner configuration. (h) Open-circuited line probe.

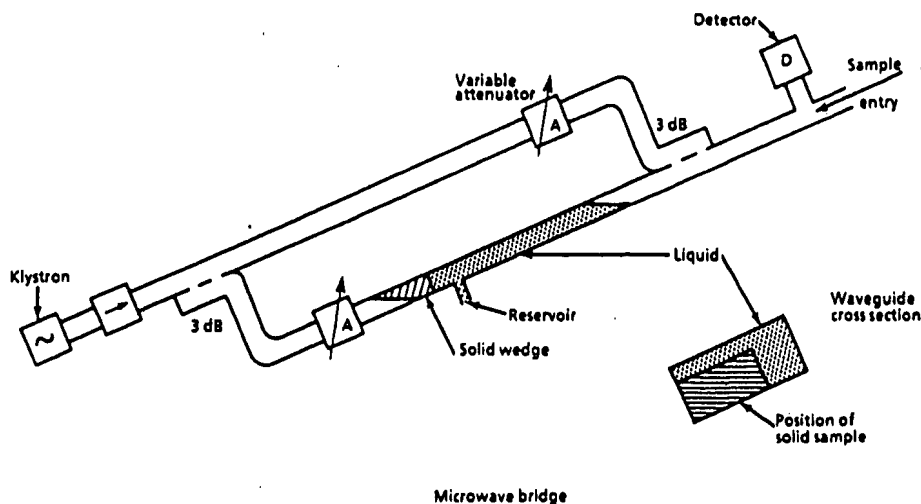


Fig. 2. Waveguide bridge for measuring low-loss liquids and solids (cf. Lynch and Ayers [64]). The tilting of the bridge (see also inset) stretches the meniscus along the waveguide to reduce discontinuity susceptances. The inset demonstrates how a solid sample can be inserted for measurement by a liquid substitution technique.

a single-pass geometry in the form of a waveguide bridge (Fig. 2), could be used for determining the loss of low-loss liquids with uncertainties comparable to those associated with cavity methods (approximately $\pm 10 \mu\text{rad}$ in $100 \mu\text{rad}$). They also measured solid dielectric samples by a liquid immersion method in the frequency range 8 to 40 GHz.

Reflection measurements are, in fact, far more popular than transmission in guided-wave geometries. The well-known Roberts and von Hippel method [65] (also [12]) is still probably the most commonly used materials measurement technique at microwave frequencies, Fig. 1 (d), (e). It has potentially become far more convenient to use with the advent of automatic network analyzers (ANAs) and, more recently, six-port reflectometers. Computer programs for rapid data reduction add to the convenience [66]. The technique is suitable for measurements over a wide temperature range: Brydon and Hepplestone [67] and Wickenden and Duerden [68] described such measurements on ceramics from 20°C to 600°C . It is also suitable for a wide range of permittivities and all but low losses, and can be modified for measuring magnetic materials (Section VI). Rzepecka and Stuchly [49] have analyzed another coaxial geometry, Fig. 1(c), in which a small sample is treated as a lumped admittance terminating the transmission line. Alternatively, the geometry of Fig. 1(b) can be simulated by terminating the line with an absorbing matched load in full contact with the material under test. In waveguide, such loads take the form of a cone or spike protruding into the guide and the resulting "modified infinite sample" geometry is suitable for reflectometric measurements on powders or liquids [69].

Guided reflectivity measurements on liquids can make full use of one's ability to vary the specimen thickness at will in order to reduce measurement error. Van Loon and Finsy [70]–[73] have described a curve-fitting waveguide technique for liquids based upon this principle, Fig. 1(f). Their technique has been applied successfully from 5 to 150 GHz.

A combined reflection/transmission approach can allow the possibility of optimizing measurements by utilizing the mutual dependence of the scattering coefficients of the

sample and its complex permittivity. Modern network analyzers are used to facilitate such measurements on dielectric samples which are placed in a transmission line between the two ports of the analyzer. One such approach is described by Weir [74] who also applies it to permeability measurement (Section VI). More recently, Lighthart [75] has presented a paper which discusses the optimization of sample length for this method and which includes a comprehensive uncertainty analysis.

D. The Use of Numerical Field Computations in Guided Geometries

Conventional guided-wave techniques assume that only one traveling-wave normal mode and its reflection are propagating at any point in the transmission medium. For this assumption to be valid, a number of constraints must be placed upon the geometries that can be used, and a severe limitation of the convenience of such techniques is often the result. The practice of using computers to numerically analyze waves propagating in more convenient sample geometries is becoming common in materials measurement. There are many examples of this approach scattered throughout this review (e.g., field computations in re-entrant cavities, TM_{01} -mode cavities, on microstrip, etc.) but the area in which this trend can best be demonstrated is that of biological dielectric measurements. Biological substances exhibit a very wide range of complex permittivity [76], [77], but most of them, being lossy, are eminently suitable for measurement by reflectometric techniques. Bianco *et al.* [78] used shielded open-circuit coaxial cells, Fig. 1(g), in which fringing electric fields from the discontinuous inner conductor extend into the dielectric under test. This gives rise to a terminating admittance which depends upon the complex permittivity of the dielectric material and the frequency. The authors analyzed the reflectivity in terms of a bilinear transform of the specimen complex permittivity, thus treating the specimen as if it were a lumped admittance. Stuchly *et al.* [79] also used this geometry for thin samples. However, computer analysis of similar geometries, as reported, for instance, by Razaz and Davies [80] for the

homogeneous case, indicates that the influence of higher order modes must be important. Corrections for the presence of such modes, which give rise to a departure from bilinearity, were employed by Bussey [81].

Numerous other geometries have been analyzed, as illustrated in Fig. 1, and these were reviewed in depth by Stuchly and Stuchly [82]. Since 1980 the open-circuit probe shown in Fig. 1(h) has become popular for noninvasive biological measurements. Its advantage is that the specimen need not necessarily be contained in a cell, and may indeed be *in vivo*. In use, fringing fields from the open circuit are again treated as a terminating admittance and measured by reflectometry. The specimen is normally assumed to be sufficiently large for effects of the back surface and sides to be ignored [83]–[89]. Numerical analyses of fringing fields for this geometry have since been supplied by Gajda and Stuchly [90] and Swicord and Davis [91]. A more complete analysis in terms of higher order TM modes [92] shows that some of the simpler approaches can lead to significant errors.

E. Microwave Closed Cavity Methods

In the face of recent improvements in transmission line techniques, the continuing popularity of resonance methods must be attributed to their greater sensitivity. The former may arguably be more convenient or more easily automated, but the "multipass" nature of resonance measurements allows more sensitive measurements to be carried out, even with smaller material specimens. The best uncertainties obtained for permittivity and loss, approximately ± 0.2 percent in a permittivity of 2 and ± 3 percent in 100 μ rad at 95-percent confidence level (Cook [93]), can indeed be equaled for some materials by transmission line measurements [64] but only by using larger samples.

Two rather different types of resonant measurements are commonly used. Perturbation methods are suitable for all permittivities, for anisotropic and magnetic materials, and for medium to high loss determinations. Their chief features are, firstly, the use of samples which are ideally very small compared with the cavity size and, secondly, the use of approximate expressions for the fields in the sample and cavity. The basic theoretical principles of this method are

well known [94], [95]. By contrast, measurements on low-loss materials use larger samples which fill a significant proportion of cavity volume. Simple geometries are preferred to allow more exact expressions for the fields to be used without the mathematics becoming too intractable.

Undoubtedly, the most popular perturbation geometry is the TM_{010} cavity, Fig. 3(a). Rod-shaped solid samples, or liquids in tubular cells, are introduced along the axis of the cavity and their properties are inferred from the change in resonant frequency and Q -factor [96]–[98]. The specimens usually extend over the complete length of the axis of the resonator, as shown in the diagram, but the measurement of frequency shift as a function of sample penetration into the cavity provides useful extra data [99]. Other resonant modes and cavity geometries can be used, as appropriate, for the materials under study, and improvements in experimental techniques for such cavities have been the concern of a number of authors [100]–[105]. Such methods are well-suited for high-temperature measurements; Ho [106] describes a perturbation method which can be used up to 1600°C at 35 GHz.

Practical difficulties and mathematical approximations can easily increase uncertainties in perturbation measurements to the order of 10 percent for permittivity. The adoption of a more complete description of the cavity fields can be afforded by employing numerical analysis; Li *et al.* [107], for example, apply such techniques to the TM_{010} cavity.

In fact, exact theories are more commonly applied to those microwave cavities which are used for measurements on very-low-loss materials (300 μ rad or less). The geometry which has proved most fruitful in the 8–40-GHz region is the TE_{01n} -mode cylindrical cavity, Fig. 3(b). The dielectric specimen in this case is disc shaped, with the same diameter as the cavity, and is ideally (for minimum uncertainty) an integral number of half-wavelengths thick [93], [108]. The requirement for removal of the degenerate TM_{11} mode has led to these cavities being constructed from helical waveguide. Cook and his co-workers showed that a sensitivity of 1 μ rad at both 10 and 35 GHz can be achieved by using high Q -factor cavities ($Q \sim 50000$) and phase-locked sources. The technique has been extended for measurements over a range of temperatures [109], [110] and for liquid dielectrics [111]. The most recent developments have been concerned

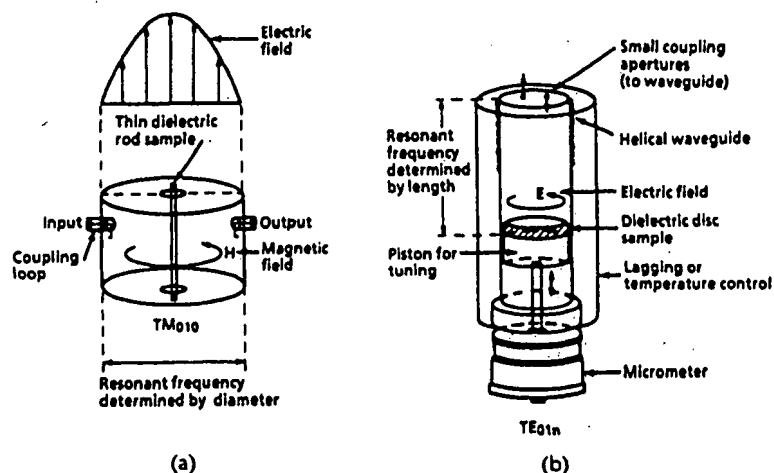


Fig. 3. Two-cavity configurations. (a) The TM_{010} cavity, commonly used for perturbation measurements up to 10 GHz. (b) The TE_{01n} cavity used for low-loss permittivity measurement (8–40 GHz).

with optimizing the cavity and sample dimensions [112], [108].

F. Open Resonators

The open resonator, Fig. 4, is a quasi-optical microwave analog of the Fabry-Perot resonator. It has a long history of use in dielectric measurements (see, for example, Culshaw [113]) and in recent years has been developed specifically as a device for measuring homogeneous low-loss materials at millimeter-wave frequencies [114]. The reader is referred to recent comprehensive reviews [115], [116] for more details of this technique and its development.

The most sensitive open resonators are of the bi-concave or plano-concave type, as shown in the diagram. Such structures exhibit TEM resonances in which the electromagnetic fields take the form of a standing-wave Gaussian

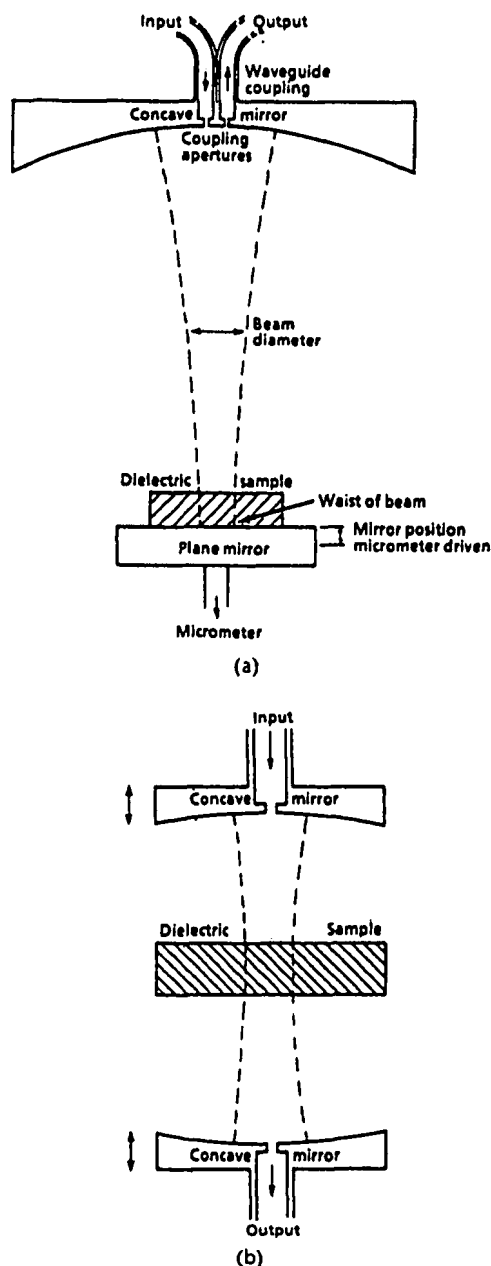


Fig. 4. Two open-resonator geometries. (a) "Hemispherical." (b) "Short." Both have been used for accurate measurements on low-loss dielectrics.

beam [117], [118]. The resonant fields have a maximum amplitude on the axis and fall away (like a Gaussian) as one moves away from it. Sample diameters down to six wavelengths are quite adequate for measurements at the "waist" of the resonator. Open resonators are commonly used in the millimeter-wave region (30–200 GHz), but they can be of value at much lower frequencies and are as sensitive as (and more convenient than) closed cavities if larger sample diameters present no problem. The Q -factors which can be achieved are remarkably high—above 150 000 at 35 GHz [114], for instance—and this property allows one to measure low losses very accurately—sensitivities down to 1 μ rad in 100 are possible. Cook *et al.* [119] intercompared measurements on unsintered PTFE and polyethylene in a TE_{01} -mode cavity and an open resonator at 35 GHz. The discrepancies obtained were as low as 0.002 in permittivity and 2 μ rad in loss.

Cullen and his co-workers developed the Gaussian-beam theory for dielectrics measurement with open resonators [118], [120], [121]. Recent reported experimental work has included anisotropy measurements [122] and accurate permittivity measurement in "short" bi-concave resonators [123]. Open resonators have many other applications [115] including magnetic materials measurement, as discussed in Section VI.

G. Microwave Free-Space Methods

Free-space methods employ beams of electromagnetic radiation which are caused to pass through the dielectric specimen. When compared to guided-wave measurements such geometries possess the advantage that the sample can be relatively easily introduced into the measurement area. Provided that the sample has flat, parallel faces, its cross-sectional shape is not important, but relatively large-aperture samples may be necessary. The open resonator, Section II-F, is one example of such a measurement geometry but all of the basic measurement principles discussed above (reflectometry transmission measurement, bridge techniques, etc.) can also be used with free-space propagation. As with guided techniques, single- or double-pass measurements are suitable for very-high-loss materials (such as RAM) and also for less lossy materials intended for use in free-space applications (such as radomes). These methods become more convenient as the frequency increases and quasi-optical propagation tends to be the norm at millimeter-wave frequencies and above as discussed in Section III-C.

Transmission measurements in which the sample is placed parallel to the wavefronts between launching and receiving antennas can easily be used to measure insertion loss and phase change of a given specimen, but such simple measurements can be enhanced in a number of ways. The incorporation of the free-space range into one arm of a waveguide bridge, Fig. 5(a), improves accuracy and, with the additional application of corrections for multiple reflections in the sample, one is able to measure the intrinsic material properties [106], [124]. Reflection measurements are also possible, Fig. 5(b) [124]. Even greater flexibility can be achieved by rotating the sample with respect to the direction of propagation. Thus Brewster angle measurements can be carried out in this way [125] or else the complex permittivity can be inferred by measuring the power transmission as a function of angle and then curve-fitting, as shown in

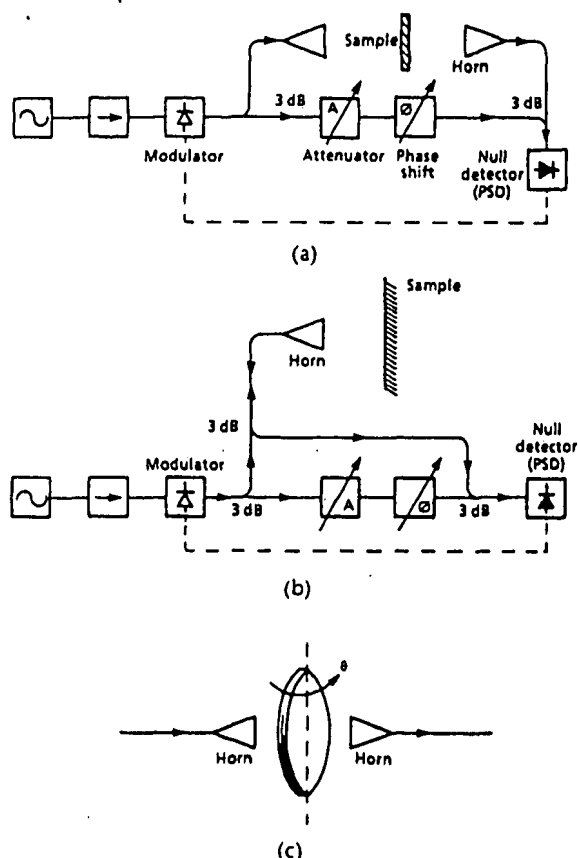


Fig. 5. Free-space measurement geometries. (a) Free-space transmission bridge. (b) Normal incidence free-space reflection measurements. (c) Measurement of power transmitted as a function of angle of incidence (cf. Shimabukuru *et al.* [127]).

Fig. 5(c), [126], [127]. This is described in greater detail in section III-C. Reflection ellipsometry should also be briefly mentioned as a free-space approach to measuring conductivity and permittivity [128].

4. Stochastic Cavity Methods

A "stochastic," "stirred-mode," or "untuned" cavity is an oversized cavity with highly reflecting walls (Fig. 6) which,

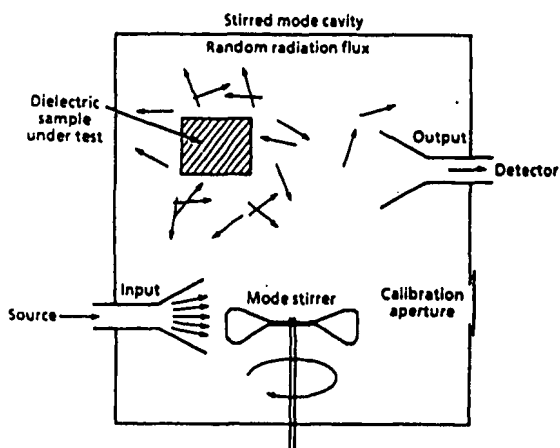


Fig. 6. "Stirred-mode" or "untuned" cavity: the mode stirrer rotates rapidly to render the internal fields homogeneous and isotropic on a time-average basis.

as a result of the action of the mode stirrer maintains throughout its volume an isotropic radiation field. Unlike the conventional, discrete-mode, resonant cavities the response to an injected electromagnetic field is relatively independent of frequency and such cavities may therefore be used as wide-band devices which in practice have applications throughout the microwave and millimeter-wave bands, in fact up to light frequencies [12]. At microwave frequencies, they were first used in the 1940s [130] for gas spectroscopy, drawing on the theory of Lamb [131], perhaps the best known example being the measurement of the line shape of the water-vapor pure rotation line near 18 GHz. However, it is only in the last decade that they have been used for solid and liquid dielectric materials. An excellent survey of the latest measurement techniques has been provided by Kremer *et al.* [132], and other aspects have been covered in reviews [8], [133].

The importance of such devices for near-millimeter-wavelength studies stems from two unique features that they possess. First, Llewellyn-Jones *et al.* [134] have shown that within certain well-defined limits, measurements can be made without placing restraints on the specimen geometry. The measurements are sensitive only to absorption loss, not to reflection or scattering losses. In other words, the homogeneous nature of a sample, inferred from the change in insertion loss of the cavity, depends to a good approximation—possibly better than 10 percent for low-permittivity materials [8]—only on its volume. Thus materials—e.g., powders—which are difficult to study quantitatively by conventional methods because of the dominance of nonabsorptive losses, become accessible to study. Using this technique, absorption measurements have been reported for a number of low-loss polymers at 156 GHz [134], for PTFE specimens between 3 and 15 cm^{-1} [135], and for polyethylene powders from 5 to 30 cm^{-1} [136].

An important advance in the technique came from the work of Kremer and Izatt [137], [138] who showed that by the use of lamellar specimens it was possible to determine both optical parameters. Basically, they take account of all interface effects and thus of the existence of standing-wave patterns in the specimen for certain frequencies. Thus from studies of several lamellae of different thicknesses it becomes possible to infer the refractive index as well as the absorption. The cavity used by Kremer and his associates has exhibited the best performance of any published to date. It is constructed from two internally dimpled hemispheres, one of which rotates rapidly with respect to the other to produce the mode-stirring action. With this equipment, measurements have been made at variable temperatures on a number of materials including PTFE, polyethylene, plexiglass, water, glycerin, and the biomolecules RNA, collagen, and lysozyme all at 70 GHz [137]–[139] and also on polyacetylene [140].

1. Microstrip and Other Guided Geometries

For a laboratory engaged in the business of producing microstrip components, it may well be that the most convenient geometry for assessing material properties is itself a microstrip configuration. One microstrip approach employs numerical analysis to obtain the intrinsic properties of the substrate itself. Pannell and Jervis [141] have described two such techniques, the "reflection canceling" method, and

the "line balancing" method. They reported 99-percent confidence-level uncertainties of ± 8 percent and ± 6 percent, respectively, for measuring permittivities of 3–4 in the frequency range 1–8 GHz. A second approach utilizes a length of microstrip as a probe or sensor for measuring the dielectric properties of a material placed uniformly over the strip and surrounding substrate. Kent [142], [143] has studied the use of such sensors for moisture measurements in the food industry. A method for testing substrate laminates, described by Traut [144], turns this "sandwich" type geometry into a stripline resonator which has advantages in sensitivity over simple microstrip single- or double-pass methods.

Examples of measurements employing other modern transmission lines are described by Itoh and Hsu [145] (inverted strip (IS) dielectric waveguide), and Jamwal and Dhar [146] who derive the permittivity of cylindrical-rod dielectric waveguide from the wavelength of standing waves measured at the surface of the rod. Unmetallized dielectric rod resonators which support their own resonant fields between metal plates at either end have also been used for dielectric measurements [147]–[149].

J. Calorimetric Measurement of Loss

The idea of making accurate estimations of dielectric loss through measuring the heat generated by the absorption of electromagnetic energy arose first in connection with cryogenic measurements on polymers below 4.2 K [150]–[152]. Measurements were extended up to 10 MHz by Gilchrist and Isnard [153]. The calorimetric technique can be very sensitive, since one is measuring directly the quantity of interest rather than inferring it from the difference of two much larger and approximately equal ones, and Carson [152], for example, reported a resolution for loss tangent as low as 5×10^{-8} . More recently, Roussy and his co-workers [145], [155] have used a calorimetric technique with granular materials at 2.45 GHz at ambient temperatures.

III. FREE-SPACE OPTICAL METHODS

A. Direct Monochromatic Methods

In the direct methods of relevance to this review the central part of the measuring equipment will be an instrument to provide the phase sensitivity necessary for the derivation of the refraction spectrum. These will either operate in free space, or may be a mixture of waveguide and optical components but with the specimen probing being performed in free space.

The instrument developed by Jones *et al.* [156] is based on a Mach–Zehnder configuration. The radiation source is a 245-GHz optically pumped laser. Most of the propagation occurs in free space, with some through low-loss circular dielectric tubes. Although the complex refraction index can, in principle, be determined from a single measurement with such an instrument, Jones *et al.* separate the refraction and absorption measurements. The former is made by effectively finding the phase shift that occurs when the specimen is removed from one arm of the interferometer. It is necessary to have either an approximate knowledge of the refractive index beforehand, or to make measurements on two different thicknesses of the same material. In the ab-

sorption measurement, the instrument is used as a radiometer to give the power transmission, and the absorption coefficient is calculated from this using the refractive index value. The uncertainty in the refraction measurements was found to vary between ± 0.0002 and ± 0.0013 for measurements on materials having refractive indices between 1.5 and 2.0. The absorption coefficients were found with uncertainties between 0.001 and 0.008 cm^{-1} , equivalent to 0.5 to 9 percent.

Kadaba [157] has recently described a largely in-waveguide system which gives both the complex relative permittivity and permeability. It operates at either 56 or 94 GHz and uses a heterodyne receiver to determine the transmission and reflection scattering coefficients of a lamella placed midway between transmitting and receiving horns. Measurements at 94 GHz on plexiglass, fiberglass, and teflon indicate random uncertainties in the real parts of the permittivity and permeability of about 5 percent, with an uncertainty nearer 10 percent in the loss tangent.

An interesting development has been the incorporation of backward-wave oscillators into spectrometers for the direct determination of permittivity and permeability [158], [159]. These spectrometers are based on Mach–Zehnder configurations [160] and have been used to measure both the complex reflection and transmission properties of specimens at wavelengths between 0.3 and 3 mm, at temperatures up to 650 K. Uncertainties in the real and imaginary parts of the permittivity were assessed at about 5 percent.

B. Direct Broad-Band Methods

A great number of measurements of the optical constants of solids from 3-mm wavelength out to about $15 \mu\text{m}$ have been made by the technique of dispersive Fourier transform spectroscopy (DFTS) (Birch and Parker [3]; Afsar [6]). This yields the full spectral variation of both optical constants of a material from a measurement of either its complex transmission or reflection coefficient. At near-millimeter wavelengths, the instruments used [8], [9] are normally the polarizing version [161]. A schematic layout is shown in Fig. 7. The source is a high-pressure mercury-vapor lamp, the detector is usually a cryogenically operated InSb bolometer, and the polarizers are made from wound wire grids. Most of the measurements have been in transmission at ambient temperature. In such a measurement, the accuracy is critically dependent on the degree of opacity of the specimen and its thickness. Under ideal conditions, a fairly transparent specimen with an optical thickness about 10 mm, it is possible to achieve random uncertainties in the refraction spectrum of less than 1 part in 10^5 , and better than $\pm 0.002 \text{ cm}^{-1}$ in the absorption spectrum. If the specimen is not plane-parallel, systematic error can dominate the derived values of the optical constants at levels in excess of the random uncertainties. Among the solids that have been studied have been alumina, beryllia, silica, borosilicate, titanium silicate, and glass ceramics together with some semiconductors, e.g., GaAs and Si (Afsar and Button [9], [162]) as well as common low-loss polymers (Birch *et al.* [7]), some commercial microwave materials (Birch [163]), and some ceramics which show promise as millimeter-wave windows (Afsar [10]). Some representative examples are shown in Figs. 8–10.

A recent development in DFTS in this spectral region has been the extension of the accessible range of measure-

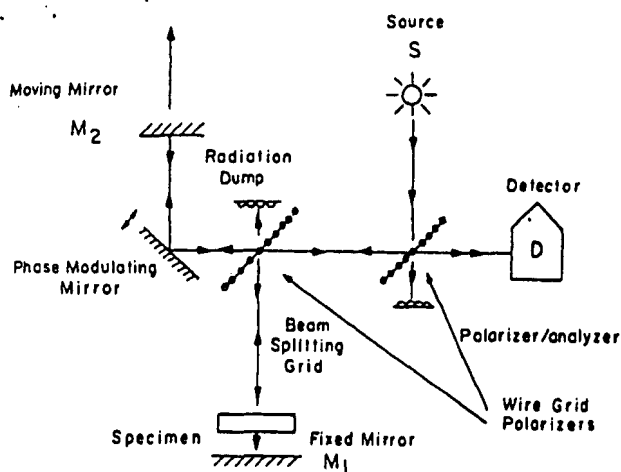


Fig. 7. Layout of a two-beam polarization interferometer for dispersive Fourier transform spectroscopy of solids. The radiation source is a high-pressure mercury vapor lamp. The polarizer/analyzer grid both polarizes the incident beam and analyzes the emergent beam before it reaches the detector. The beam-splitter grid splits the polarized beam and later recombines the two partial beams [10].

ments in transmission to include any temperature between 4.2 and 300 K [164]. In such measurements, it is necessary to know the thermal contraction of the specimen over the measured temperature range in order to know its thickness at each temperature. Such information is not often available for new materials and the DFTS method has been extended to give the thickness information as well as the optical constants [165].

C. Indirect Monochromatic Methods

These rely on measuring various combinations of the power transmission and reflection coefficients of a lamella as functions of radiation frequency, angle of incidence, and orientation of the polarization. In the system of Kinasewitz and Senitzky [166] the power transmission and the reflection coefficients of a lamella are measured at 17° incidence at 107.3 GHz for plane-polarized radiation incident perpendicular to the plane of incidence. The radiation was launched at the specimen by a horn antenna and coupled back into waveguide by a similar horn for direct detection. The same receiver was used for transmission and reflection studies. A number of silicon specimens of different resistivities were studied. The complex permittivity of each was calculated using a numerical solution of the equations describing the transmission and reflection properties of a lamella. Measurement accuracies were not explicitly mentioned. Shimabukuro *et al.* [127] describe a different indirect technique in which the power transmission of a lamella is measured as a function of angle of incidence between 0 and 50° , see Fig. 5(c). This was done at 93.8 GHz again using plane-polarized radiation incident perpendicular to the plane of polarization as this gave enhanced accuracy in the real part of the permittivity over that which would have been achieved with parallel polarization. The specimen was mounted in free space between transmitting and receiving horns, the detection again being in waveguide. The complex permittivity was derived from the measurements using a best fit bootstrap estimate method. Results were reported for low-loss materials such as teflon, Rexolite, TPX, fused

quartz, and several casting resins. The permittivities of these varied between 2 and 5.7, with standard errors between 0.004 and 0.009. The corresponding values of the loss tangent varied between 0.00021 ± 0.00003 and 0.0042 ± 0.0001 .

Rachford and Forester [167] describe a system using backward-wave oscillators between 75 and 110 GHz. The power transmission and reflection of a lamella are measured as a

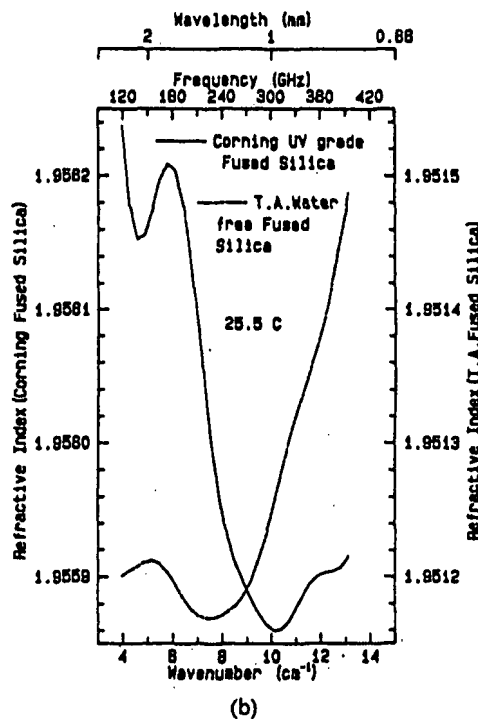
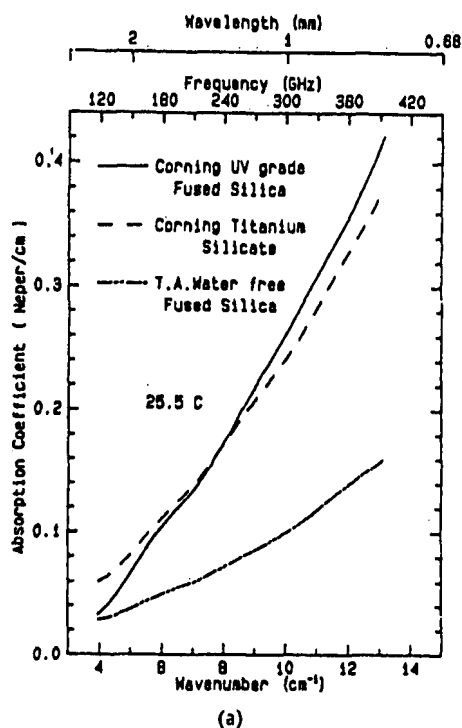


Fig. 8. (a) Absorption spectra of Corning UV grade fused silica (water content 1200 ppm), Corning titanium silicate (7 percent by weight T102), and Thermal American water-free fused silica (water content 5 ppm). (b) Refractive index spectra at ambient temperature of two of the samples from (a). These spectra reveal well-resolved and highly characteristic features at low wavenumbers.

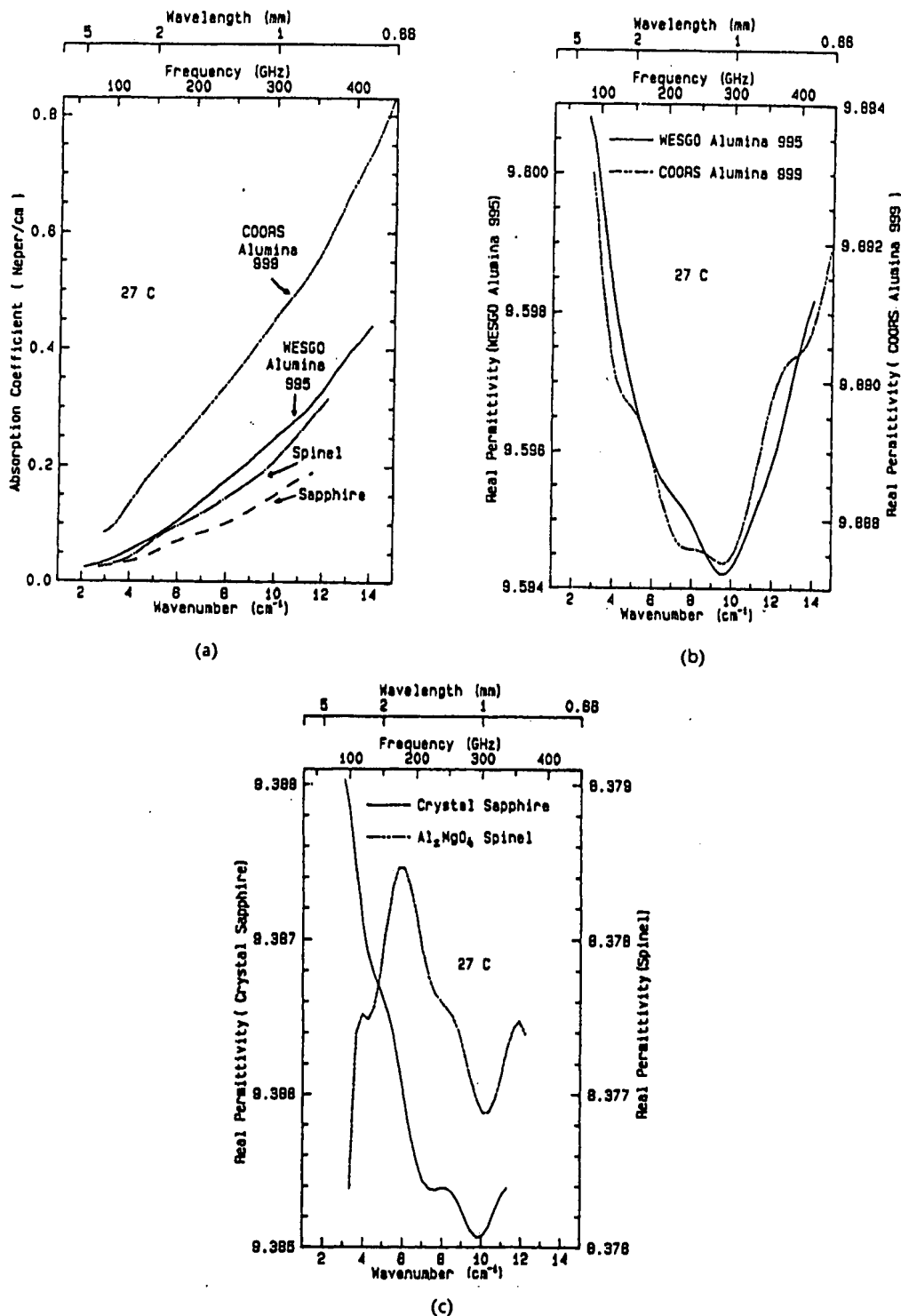


Fig. 9. (a) Absorption spectra of COORS alumina 999, WESGO alumina 995, COORS hot-pressed MgAl_2O_4 spinel, and Crystal Systems Inc. single-crystal Z-cut sapphire all at 27°C. (b) Refraction spectra for two of the samples from (a). (c) Refraction spectra for the remaining two specimens.

function of frequency for various angles of incidence at the two prime polarization orientations. The recorded data can be analyzed in either of two ways to find the complex permittivity and permeability. One involves a least squares fit to the data at discrete frequencies across the measured spectral range. The other involves a least squares fit to all the data assuming line-shape functions for the frequency behavior of both permittivity and permeability. The first of these two is the more general method, and in an analysis of

measurements on lithium ferrite discs the random uncertainties in the real and imaginary parts of the complex permittivity typically appear as 0.05 and 0.01, respectively. This is in a region where the real part was about 16.0 and the imaginary one 0.5. When the second analysis is used these uncertainties decrease by at least an order of magnitude, but this requires prior assumptions about line shape. If this technique is used on magnetic materials it is necessary that some measurements are made on thin specimens

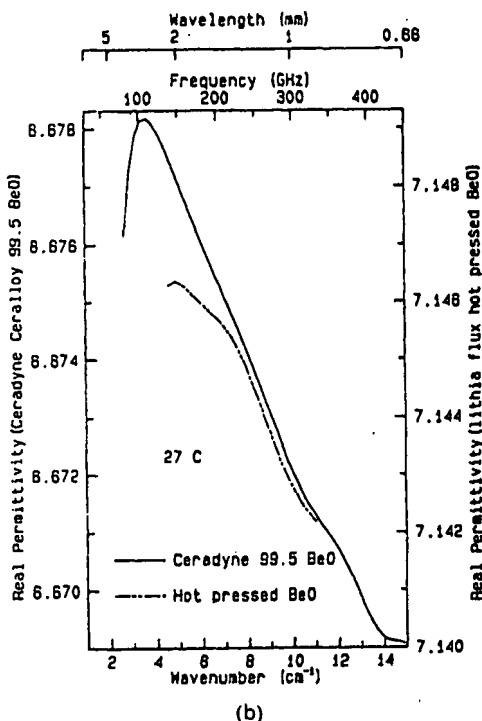
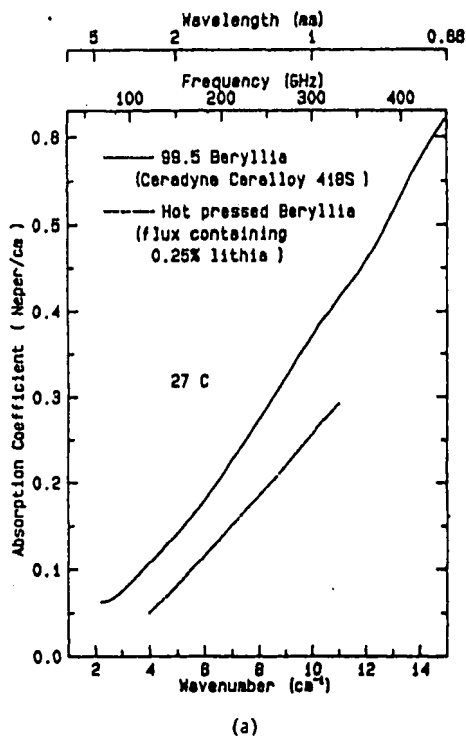


Fig. 10. (a) Absorption spectra of two types of pressed beryllia, the hot-pressed form has the higher crystallinity and the lower absorption coefficient. (b) Refraction data for the same samples.

D. Indirect Broad-Band Methods

The broad-band indirect methods in use are mostly based on FTS [2], [168]. Some do, however, involve the use of approximate expressions and some require assumptions to be made about the refractive index [169]. A method that is free of these limitations is the use of the channeled spectra which arise as a consequence of interference between the internally reflected rays of a lamella. The method has been

widely used and discussed. Randall and Rawcliffe [170], for example, show how the refractive index can be calculated from the period of a spectrum using a rapidly converging technique to calculate the reflection phase shift. The absorption coefficient is then found from a low-resolution spectrum in which the channel spectrum is and is not resolved, the period of the channel spectrum being found from the positions of the zero crossings of the difference spectrum. These can be located more accurately than the maxima and minima of channel spectrum, as traditionally used.

Simonis and co-workers [172], [173] have applied this technique at near-millimeter wavelengths, presenting measurements on a variety of materials including crystal quartz, Rexolite, and a number of beryllia ceramics. In these measurements, the uncertainty in the refractive index was less than 0.05 percent if the specimen thickness was known to be better than this. Typical levels of uncertainty in the absorption coefficient were quoted as ± 10 percent of the maximum value, or $\pm 0.05 \text{ cm}^{-1}$, whichever was greatest.

IV. IMPROVING ACCURACY—MEASUREMENT INTERCOMPARISONS

The effect upon a given measurement method of its inherent random errors can always be reduced to an arbitrarily small amplitude by averaging the results of a sufficiently large number of determinations but, clearly, this will have no effect on the systematic errors. One can only hope to eliminate the latter by comparing results from widely differing measurement methods. For this reason, intercomparison of measurements, especially of those based on different principles, has always been a valuable means of improving the quality of metrology. However, apart from the "in-house" intercomparisons mentioned above (Section II), formal dielectric-measurement intercomparisons have been reported only at infrequent intervals [174], [175]. Now that so many new methods have been added to the dielectricians armor, this is an aspect of materials metrology that should perhaps be given more prominence.

V. SOME TYPICAL EXAMPLES OF HIGH-PRECISION MILLIMETER/SUBMILLIMETER-WAVE MEASUREMENTS

As mentioned earlier, millimeter/submillimeter-wave measurements are now being made worldwide on an enormous range of substances for both scientific and technological purposes. However, measurements at the "state-of-the-art" level of precision tend to be restricted to just a small number of laboratories and, naturally at these laboratories, to be carried out mostly on the technologically more important materials. These would be those suitable for making lenses, windows, dielectric waveguides, etc., and those used in the manufacture of high-speed integrated circuits and for the substrate in microstrip circuits. In these connections it is now very important to be able to detect variations between nominally equivalent samples so that preparative procedures can be developed which can reliably give a high-level of reproducibility.

Classes have always been attractive as optical materials, not just because of their transparency, but also because their optical parameters can be continuously varied by judicious adjustments to their stoichiometries. Unfortunately, the good transparency is mostly restricted to the visible and

near-visible regions and in most other spectral regions glasses are less useful. However, below about 10 cm^{-1} many glasses regain moderate transparency and silica, especially the crystalline form, quartz, transmits well at even higher frequencies. Some examples, due to Afsar, are shown in Fig. 8. These demonstrate how important water content is in determining the absorption—the heavy ion content being much less significant. The high precision now available in refractive index measurements [10], [11] permits the observation of discrete features in the refraction spectrum at very low wavenumbers. Similar features have been reported by Birch and his colleagues [176]. Ceramics based on beryllia or alumina are very valuable for high-temperature work: alumina can be used as high real permittivities, which enable them to be used in dielectric waveguide construction. Fig. 9 shows some results on four ceramics based on Al_2O_3 . The "WESGO" alumina 995 and the "COORS" alumina 999 were cold-pressed then sintered. It is most interesting to see how nominally equivalent materials can have such different properties. In the present case, it is very remarkable that the notionally purer material has the higher loss. The MgAl_2O_4 spinel sample was also made by the COORS Porcelain Co. The still lower loss in this material is probably due to the higher degree of crystallinity which came about during the hot pressing. When it comes to the highly crystalline sapphire (Crystal Systems Inc.) the losses are found to be even lower. These spectra indicate that DFTS could prove to be a very useful tool in the monitoring of materials processing [10].

The absorption and the real part of the permittivity for two kinds of beryllia (BeO) are shown in Fig. 10(a) and (b). The higher degree of crystallinity which has been introduced into the Union Carbide BeO has obviously significantly reduced the absorption coefficient (by some 43 percent) compared with the cold-pressed "Ceradyne" Ceralloy 4185 99.5 beryllia. In practical applications, the advantage of using a hot-pressed window, rather than a single-crystal window is that it is not brittle in high-temperature conditions. One might then suggest the use of specially prepared "spinel" or hot-pressed BeO as a window material for kilowatt/megawatt CW gyrotrons. The permittivity data for both cold- and hot-pressed BeO show similar behavior except that the values are higher for the hot-pressed version, as expected from the high degree of crystallinity [10].

Semiconductors are extremely valuable, in fact one could say irreplaceable, submillimeter materials but it is clear that in those applications which require transparency, very-high-resistance samples are needed. The basic reason for this is that free-carrier absorption tends to dominate in the submillimeter region and one, therefore, needs to reduce the concentration of free carriers if one is to reduce the absorption. Free carriers can be suppressed by going to high-purity material but one can also achieve the same effect in less pure material by deliberate doping with a "deep-trap" impurity. Chromium is a very suitable deep trap for GaAs and Cr-doped substrates are now available. For example, two Cr-doped high-resistivity ($\rho \sim 10^7\ \Omega \cdot \text{cm}$) GaAs samples have been measured. One was obtained from the Hughes Aircraft Corporation (Cr concentration of $5 \times 10^{15}/\text{cm}^3$, $\rho \sim 5 \times 10^7\ \Omega \cdot \text{cm}$). Fig. 11(a) compares their absorption coefficient spectra. The "Hughes" specimen with higher Cr concentration shows almost a "near-plateau" from 70 to 250 GHz. The rapid increase of the absorption at higher frequencies reflects the tail of the 600-GHz multi-

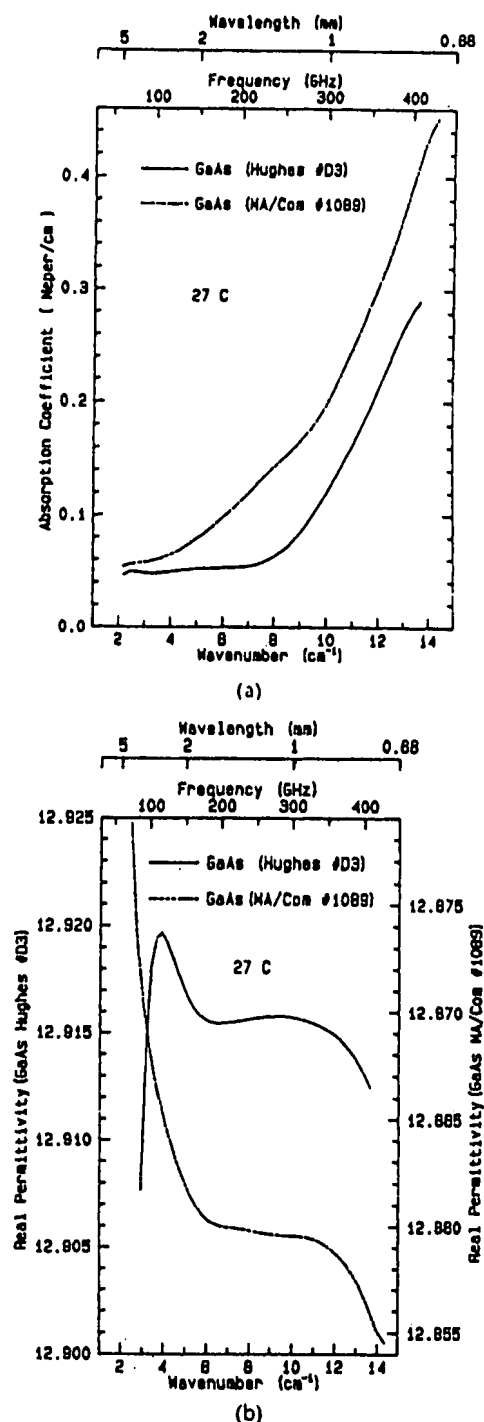


Fig. 11. Comparison of absorption spectra of Hughes and MA/Com chromium-doped single-crystal high-resistivity ($\rho < 10^7\ \Omega \cdot \text{cm}$) samples. (a) The rise in absorption with frequency is due to a multiphonon absorption peak centered near 600 GHz. The higher chromium and hence "deep-trap" concentration in the Hughes sample has, by suppressing the free carriers, led to lower absorption at the millimeter-wave end of the spectrum. (b) Refraction data for the same samples.

phonon absorption band [177]. The data for the real part of the dielectric permittivity are shown in Fig. 11(b) for both GaAs specimens [10].

VI. MEASUREMENTS ON MAGNETIC MATERIALS

There are a number of reasons why the characterization of magnetic materials is a more complex problem than that

of pure dielectrics. Both their permittivity and permeability have to be determined for microwave applications, and the latter is likely to be a tensor quantity. Nonlinearity and hysteretic behavior have to be taken into account in nearly all applications—phenomena which are much less important for commonly used microwave dielectrics. Unlike pure dielectrics, which generally exhibit only broad relaxations at microwave frequencies, ferrites exhibit sharp ferrimagnetic resonances (FMRs) which have important applications in isolators, circulators, modulators, etc. (see for example Abeyrie *et al.* [178]). In spite of these difficulties, the instrumental solutions to the practical problem of measuring magnetic materials very often turn out to be similar to those used for pure dielectrics. They have, however, the added complication of one's having to apply static magnetic fields (where appropriate) to the region around the sample cell.

Many approaches have been adapted for magnetic materials, a few examples must suffice here. A number of transmission line methods have been reported. Weir [74] described such a technique which makes use of both reflection and transmission coefficients measured on an ANA. More recently, Josyulu *et al.* [179] compared a reflectometric method using a large number of sample widths with the Roberts and von Hippel method [65]. In the latter, both permittivity and permeability of a sample are measured by placing it before short and open circuits (in practice, short and offset short). Faraday rotation measurements have also been used for determining tensor permeability of ferrites at 0.75 GHz (Ito *et al.* [180]).

Resonance methods are popular. Waldron [95] has described a basic theory for using perturbation techniques to measure ferrites, but a number of other approaches for magnetic (including tensor) properties in closed cavities have been developed more recently [181]–[184]. At millimeter-wave frequencies open resonators have proved to be of value in assessing FMRs of ferrite materials [185]–[187]. Two millimetric techniques for measuring permeability [158], [159] are discussed in Section III-A.

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